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(54) WET TYPE TONER AND MANUFACTURE THEREOF

(57)Abstract:

PURPOSE: To prevent the picture flow from occurring by dispersing synthetic resin particles contg. no additive or those contg. a coloring agent in a liquid aliphatic hydrocarbon and adding inorganic particulate matter to the liquid.

CONSTITUTION: The electrical insulating liquid aliphatic hydrocarbon having ≥1010Ω.cm volume resistivity is used to enhance the electrical insulation properties of the wet type toner. Also this hydrocarbon has only small dissolving power to olefin resins and therefore the toner is prevented from being deteriorated by using the hydrocarbon. As this liquid aliphatic hydrocarbon, n-paraffin hydrocarbons, etc., are suitable and branched chain aliphatic hydrocarbons are particularly preferred. As the synthetic resin particles, particles of a olefin resin having a carboxyl or ester group and also the presence of a polyhydroxycarboxylic ester having a trimer to decamer of hydroxycarboxylic ester monomer in the liquid apliphatic hyudrocarbon are desirable. Examples of the inorganic particulate matter are silicon dioxide, aluminum oxide, etc.

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CLAIMS

[Claim(s)]
[Claim 1] The wet toner characterized by making the non-subtlety particle fine particles for image flow prevention contain in the wet toner which has the synthetic-resin particle which added synthetic-resin particle independence or a coloring agent, and liquefied aliphatic hydrocarbon.
[Claim 2] The wet toner according to claim 1 which a synthetic-resin particle is an olefin system resin particle which has a carboxyl group or an ester group, and is characterized by making the polyhydroxy carboxylate of 3 which makes hydroxycarboxylic acid a monomer - 10 **** exist in liquefied aliphatic hydrocarbon.
[Claim 3] Claim 1 characterized by non-subtlety particle fine particles consisting of an oxide, or a wet toner given in two.
[Claim 4] the manufacture approach of the wet toner characterize by make the polyhydroxy carboxylate of 3 - 10 **** and non-subtlety particle fine particles which make hydroxycarboxylic acid ester a monomer exist in the production process in give a mixed distribution process and manufacture a wet toner after mix with liquefied aliphatic hydrocarbon, inherit and cool under warming of the olefin system resin particle which have a carboxyl group or an ester group, and a coloring agent.
[Claim 5] Are and the heating dissolution is carried out at the high solvent of temperature dependence. solubility [as opposed to this resin for the olefin system resin and the coloring agent which have a carboxyl group or an ester group] -- After considering as the resin solution which the coloring agent distributed, while throwing in this resin solution in liquefied aliphatic hydrocarbon, cooling and depositing a resin particle In this liquefied aliphatic hydrocarbon's permuting a solvent and manufacturing a wet toner, it sets in the production process. The manufacture approach of the wet toner characterized by making the polyhydroxy carboxylate of 3 - 10 **** and non-subtlety particle fine particles which make hydroxycarboxylic acid ester a monomer exist.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the wet toner which was suitable as the object for electrophotography, the object for electrostatic printing, and an object for information record, and its manufacture approach about the wet toner which develops an electrostatic latent image with the toner distributed in the insulating liquid, and its manufacture approach.
[0002]
[Description of the Prior Art] An electrostatic latent image develops, a transferred object is stuck to the photo conductor front face to which the toner adhered, direct-current corona discharge of reversed polarity performs with the charge of a toner from the tooth back of a transferred object, the approach of carrying out a suction imprint is in a transferred object about a toner, and it is adopted as the formation approach of the image which imprints the electrostatic latent image which exposed and formed the photo conductor which consists of an optical semi-conductor charged electrostatic by light to a transferred object in the dry-type copying machine with an insulating dry-type toner.
[0003] While equipment is simple, in order for this approach to hold the corona discharge charge given from a transferred object tooth back good at a transferred object tooth back and not to cause the discharge at the time of separation It is necessary to hold the electric resistance of a transferred object within the limits of 109-1012-ohmcm, and there is a fault which is easy to be influenced of the content moisture of paper etc. when environmental humidity and a transferred object are the things of absorptivity like paper, and turbulence, a greasing, etc. of a toner image pose a problem at the time of an imprint.
[0004] On the other hand, since a toner particle is in an electric insulation liquid in a wet toner, it is hard to be influenced of humidity, and it has the advantage that there are no turbulence and greasing of the toner image at the time of an imprint.
[0005] however , while it have an advantage , like the wet toner which distributed the resin particle have good imprint effectiveness , and cleaning fitness be good , the charge regulator which use in order for that image flow arise to pose a problem since the particle cohesive force at the time of the approach set in the development of a toner particle be small , and to control the charge in the inside of the liquid of a toner serve as superfluous ion , and also reduce the electric resistance of an electric insulation liquid remarkably cause image flow .
[0006]
[Problem(s) to be Solved by the Invention] Its particle cohesive force at the time of approach grouping in the development of a toner particle is large, and this invention offers a technical problem the wet toner with which toner physical properties, such as image flow with which the electric resistance of an electric insulation liquid is not reduced, have been improved, and its manufacture approach while the resin particle of a submicron unit is obtained and it is made to the narrow thing of particle size distribution, without using tumbling media.
[0007]

[Means for Solving the Problem] The wet toner of this invention is a wet toner which made the non-subtlety particle fine particles for image flow prevention contain in the wet toner which distributed the synthetic-resin particle containing a synthetic-resin particle independent or a coloring agent in liquefied aliphatic hydrocarbon.

[0008] Moreover, the non-subtlety particle fine particles for image flow prevention are made to exist in the olefin system resin particle which has the cull BOSHIKIRU radical or ester group which added the olefin system resin particle independent or the coloring agent which has a cull BOSHIKIRU radical or an ester group as a synthetic-resin particle, and the wet toner which made the polyhydroxy carboxylate of 3 which makes hydroxycarboxylic acid ester a monomer - 10 **** exist in liquefied aliphatic hydrocarbon.

[0009] First, the constituent in the wet toner of this invention is explained. The liquefied aliphatic hydrocarbon which is an electric insulation liquid has the volume resistivity of 1010 or more ohm-cm, and it is used for the purpose of raising the electric insulation in a wet toner, and it is required that solvent power should be comparatively small to olefin system resin, and, thereby, degradation as a wet toner is prevented.

[0010] As liquefied aliphatic hydrocarbon, liquefied n-paraffin hydrocarbon, iso-paraffin hydrocarbon or its mixture, halogenated aliphatic hydrocarbon, etc. are mentioned. It is desirable to be branched chain aliphatic hydrocarbon preferably, for example, to use Isopar G, Isopar H, Isopar K, Isopar L, Isopar M, Isopar V, etc. by the exon company especially. These hardly have solubility to ethylene-vinyl acetate copolymerization resin, and the solubility differences (25 degrees C and 65 degrees C) of the solubility of resin [as opposed to Isopar H] are below 0.001g / solvent ml. At the time of wet toner preservation, liquefied aliphatic hydrocarbon has [it is good to make 0.1 - 50 % of the weight exist preferably 0.01 to 80% of the weight, and / saving, after having been condensed by this solid part concentration] aging on the basis of the total weight of a wet toner, and is desirable. In addition, it is good to dilute and use it with liquefied aliphatic hydrocarbon so that solid part concentration may become 0.5 - 2 % of the weight as a wet toner at the time of development, and thereby, desirable printed matter is obtained.

[0011] As olefin system resin, an ethylene-vinylacetate copolymer is desirable. As an ethylene-vinylacetate copolymer, when it mentions by the trade name, he is TOSOH. Make URUTORASEN Series, For example, 510X, 515F, 530,537,537L, 537S, 525,520F, 540,540F, 541,541L, 625,630,630F, 682, 627, 631, 633, 680, 681, 635, 634, 710, 720, 722, 725, The 751,750,760th grade, Sumitomo Chemical Make Sumi Tait series, for example, DD-10 and HA- 20, HC-10, HE-10, KA-10, KA-20, and KA- 31, KC-10, KE-10, MB-11, and RB-11 etc. -- Mitsui and DEYUPON poly chemical Make Eve FREX series, 45X, Y-W, and 150, 210, 220, 250, 260, 310, 360, 410, 420 and 450,460,550,560 etc. -- [for example,] Japanese composition industrial SOAGUREN series -- for example, -- BH, CH, CI, DH, etc. this SOAREKKUSUSHIRIZU -- for example, -- The Takeda Chemical Industries, Ltd. DEYUMIRAN series D-219, for example, DEYUMIRAN, such as RBH, RCH, and RDH, D-229, D-251S, C-2280, C-2270, C-1590, C-1570, and C-1550 grade are mentioned. Moreover, Mitsubishi Petrochemical ERUPAKKUSU by Make YUKARON-Eve and U.S. Du Pont etc. can be used.

[0012] In addition, if the thing and an example which denaturalized polyolefin resin and introduced the carboxyl group are given by the trade name Nippon Oil chemistry Make N Polymer, TONEN petrochemistry Make TONEN CMP-HA series, Mitsubishi Petrochemical Make MODIC, Seitetsu Kagaku Make ZAIKUSEN, Mitsui Toatsu Chemicals Make ROMPURAI, Mitsui Petrochemical Industries Make ADOMA etc., When it mentions by the copolymer of ethylene and an acrylic acid, and the trade name, moreover, the Dow-Jones EAA copolymer by the Dow Chemical Co., Mitsubishi Petrochemical Co., Ltd. YUKARON EAA, Mitsui and DEYUPON poly chemical NYUKURERU, Further Sumitomo Chemical Co., Ltd. Acryft etc. A copolymer with ethylene, an acrylic acid, or methacrylic acid, When it mentions by the so-called ionomer over which they were made to construct a bridge further, and the trade name, or Surlyn by U.S. Du Pont, Mitsui and DEYUPON poly chemical Co., Ltd. -- make -- yes, if it mentions by EVAby BASF A.G. 1 wax addition, such as milan and a KOBOREN latex by Asahi Chemical Co., Ltd., and the partial saponification object of the copolymer of ethylene and vinyl acetate, and the trade name Takeda Chemical Industries The copolymer of ethylene,

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such as Make DEYUMIRAN, and acrylic ester, If it mentions by the trade name, Nippon Unicar DPD-6169 grade, the polyolefine system resin which contains the carbonyl group of carboxyl nature further can be mentioned, and one sort or two sorts or more can be used for these resin, mixing.
[0013] Next, polyhydroxy carboxylate is explained. When polyhydroxy carboxylate is made to exist in a resin particle formation process, it has a granulation adjustment function, and the resin particle to which particle size distribution were equal is obtained. Moreover, a resin particle has the structure top compatibility, and functions also as a dispersant.
[0014] Let the carboxylic acid in the hydroxycarboxylic acid the hydroxycarboxylic acid ester which is the polymerization raw material of polyhydroxy carboxylate is indicated to be by the bottom formula be alkyl or the thing by which aralkyl esterification was carried out and the amidated thing, or a metal salt. Formula HO-X-COOH (X is the aliphatic hydrocarbon of the divalent saturation containing the carbon atom of at least 12, or partial saturation, or divalent aromatic hydrocarbon containing at least six carbon atoms, and there are at least four carbon atoms between a hydroxy group and a carboxyl group among a formula.)
As such hydroxycarboxylic acid, for example A ricinoleic acid, 10-hydroxy stearin acid, 12-hydroxy stearin acid, the hydrogenated castor oil fatty acid (stearin acid little to 12-hydroxy stearin acid, and palmitic-acid inclusion), 16-hydroxy hexadecanoic acid, a 15-hydroxy pentadecane acid, 12-hydroxy dodecanoic acid, 4-hydroxybenzoic acid, a 2-hydroxy-1-naphthoic acid, A 3-hydroxy-2-naphthoic acid, a 1-hydroxy-2-naphthoic acid, 2-hydroxyphenyl acetic acid, 3-hydroxyphenyl acetic acid, 4-hydroxyphenyl acetic acid, and 3 -(4-hydroxyphenyl)- It is a propionic acid etc.
[0015] Moreover, as a derivative of hydroxycarboxylic acid, the metal salt of hydroxycarboxylic acid, such as hydroxy carvone alkyl ester, such as 12-methyl-hydroxystearate ester and 12-hydroxy stearin acid ethyl ester, 12-hydroxycarboxylic acid lithium, and 12-hydroxycarboxylic acid aluminum, and hydroxycarboxylic acid AMAIDO, hydrogenated castor oil, etc. are mentioned, for example.
[0016] By carrying out the partial saponification of the above-mentioned hydroxycarboxylic acid ester under existence of little amines or a catalyst, the polymerization of the polyhydroxy carboxylate is carried out, it is obtained, and takes various gestalten, such as a thing depended on esterification between molecules as the polymerization gestalt, and a thing to depend on esterification by intramolecular. The polymerization degree in the polyhydroxy carboxylate in this invention has desirable 3 - 10 ****, and it is the wax-like matter of light ashes brown. If the polymerization degree is smaller than 3 or larger than 10, even if there are not liquefied aliphatic hydrocarbon and compatibility and it uses it for a granulation process, the particle size distribution of a resin particle will be large, and an expected thing will not be obtained. Although especially the addition of polyhydroxy carboxylate is not limited, it is used at 0.01 % of the weight - 200% of the weight per resin weight of a rate.
[0017] Next, as a coloring agent, a well-known organic or inorganic coloring agent can be used. As a coloring agent of a black system, the carbon black of an inorganic system, a tri-iron tetraoxide, and the cyanine black of an organic system are mentioned. As a yellow system coloring agent, the chrome yellow of an inorganic system, cadmium yellow, Synthetic Ochre, Titanium Yellow, ochre, etc. are mentioned. moreover, as an acetoacetanilide system monoazo pigment of a poorly soluble metal salt (azo lake) Hansa yellow G (C.I.No.pigment Yellow 1 and the following) Similarly Hansa yellow 10G (pigment Yellow 3), Hansa yellow RN (pigment Yellow 65), HANZA brilliant-yellow 5GX (pigment Yellow 74), HANZA brilliant-yellow 10GX (pigment Yellow 98), The permanent yellow FGL (pigment Yellow 97), Simla lake fast yellow 6G (pigment Yellow 133), As RIO Nor Rui Heroux K-2R (pigment Yellow 169) and an acetoacetanilide disazo pigment Diarylide Yellow G (pigment Yellow 12), Diarylide Yellow GR (pigment Yellow 13), Diarylide Yellow 5G (pigment Yellow 14), Diarylide Yellow 8G (pigment Yellow 17), Diarylide Yellow R (pigment Yellow 55), and the permanent yellow HR (pigment Yellow 83) are mentioned.
[0018] As a disazo condensation pigment, chromophthal yellow 3G (pigment Yellow 93), chromophthal yellow 6G (pigment Yellow 94), and chromophthal yellow GR (pigment Yellow 95) are mentioned. Furthermore, as a bends imidazolone system monoazo pigment, HOSUTA palm yellow H3G (pigment Yellow 154), HOSUTA palm yellow H4G (pigment Yellow 151), HOSUTA palm yellow H2G (pigment

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Yellow 120), HOSUTA palm yellow H6G (pigmentYellow 175), and the HOSUTA palm yellow HLR (pigment Yellow 156) are mentioned. As an isoindolinone system pigment, moreover, IRUGA gin yellow 3RLTN (pigment Yellow 110), IRUGA gin yellow 2RLT, IRUGA gin yellow 2GLT (pigment Yellow 109), The fast gene super yellow GROH (pigment Yellow 137) The fast gene super yellow GRO (pigment Yellow 110), Sandrine yellow 6GL (pigment Yellow 173) is mentioned. In addition, the flavanthrone which is the Indanthrene system pigment (pigment Yellow 24), Anthra millimeter MIJIN (pigment Yellow 108), Phthloyl amide mold anthraquinone (pigment Yellow 123), HERIOFASUTOIEROE3R (pigment Yellow 99), The azo system nickel complex pigment which is a metal complex pigment (pigment Green10), A nitroso ** nickel complex pigment (pigment Yellow 153), An azomethine system copper complex pigment (pigment Yellow 117), the phthalimide kino FUTARON pigment (pigment Yellow 138) which is a kino FUTARON pigment further are mentioned.
[0019] Moreover, as a Magenta system coloring agent, the cadmium red of an inorganic system, red ocher, vermilion, a minium, and antimony vermilion are mentioned. As an azo lake system of an azo system pigment, moreover, brilliant carmine 6B (pigment Red57:1), Lake Red (pigment Red53:1) and Permanent Red F5R (pigment Red48), Lithol Red (pigment Red49), Persia Orange (pigment Orange17), A clo SEIO range (pigment Orange18), the HERIO orange TD (pigment Orange19) Pigment scarlet (pigment Red60:1), Brilliant scarlet G (pigment64:1), the HERIO red RMT (pigment Red51), Bordeaux 10B (pigment Red63) and helio bordeaux BL (pigment Red54) are mentioned. moreover, as an insoluble azo system (monoazo, a JISUAZO system, condensation azo system) Para Red (pigment Red1), Lake Red 4R (pigment Red3), Permanent Orange (pigment Orange5), Permanent Red FR 2 (pigment Red2) Permanent Red FRLL (pigment Red9), Permanent Red FGR (pigment Red112), Brilliant carmine BS (pigment Red114), permanent carmine FB (pigment Red5), P. V. carmine HR (pigment Red150), permanent carmine FBB (pigmentRed146), Nova palm red F3 RK-F5RK (pigment Red170), The nova palm red HFG (pigment Orange38), Nova palm red HF4B (pigment Red187), nova palm orange HL.HL-70 (pigment Orange36), P. V. carmine HF4C (pigment Red185), HOSUTABAMUBURAUN HFR (pigment Brown25), Balkan Peninsula Orange (pigment Orange16), pyrazolone Orange (pigment Orange13), Pyrazolone red (pigment Red38) is mentioned. Further CROMOPHTAL Orange 4R (pigment Orange31), CROMOPHTAL Scarlett R (pigment Red166), and the CROMOPHTAL red BR (pigment Red144) are mentioned as a disazo condensation pigment.
[0020] As an anthraquinone pigment which is a condensation polycyclic pigment, moreover, pyran SURON Orange (pigment Orange40), Anthanthrone Orange (pigment Orange168), JIANTORA kino nil red (pigment Red177) is mentioned. As a thioindigo system pigment, a thioindigo Magenta (pigment Violet38), Thioindigo violet (pigment Violet36), Thioindigo red (pigment Red88) is mentioned and a peri non orange (pigment Orange43) is mentioned as a peri non system pigment. Further as a perylene system pigment Perylene red (pigment Red190), perylene Vermillion (pigment Red123), Perylene MARUN (pigment Red179), perylene Scarlett (pigment Red149), Perylene red (pigment Red178) is mentioned. As a Quinacridone system pigment The Quinacridone red (pigment Violet19), The Quinacridone Magenta (pigment Red122), Quinacridone MARUN (pigment Red206), Quinacridone Scarlett (pigment Red207) is mentioned, in addition a pyrrocoline system pigment, a red system Fluor Bin system pigment, and a blue-and-white porcelain lake system pigment (water-soluble-dye + precipitant -> lake-ized fixing) are mentioned as a condensation polycyclic pigment.
[0021] As a cyanogen system coloring agent, the ultramarine blue of an inorganic system, Berlin blue, cobalt blue, cerulean blue, etc. are mentioned. As a phthalocyanine system First gene bull-BB (pigment Blue 15), SUMITON cyanine blue HB (pigment Blue 15), The cyanine blue 5020 (pigment Blue 15:1), SUMIKA print cyanine blue GN-O (pigment Blue 15), The fast sky blue A-612 (pigment Blue 17), Cyanine Green GB (pigment Green7) and cyanine Green S537-2Y (pigment Green36), The SUMITON fast violet RL (pigment Violet23) is mentioned. Moreover, the Methyl Violet Lynn molybdic-acid lake (PV-3) which is the indanthrone blue (PB-60P, PB-22, PB-21, PB-64) and the basic-dye lake pigment which are the Indanthrene system pigment is mentioned. In addition, the coloring agent called the so-called processing pigment which coated the front face of the above-mentioned coloring agent with resin can be used similarly.

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[0022] Moreover, when the transparency of the image when forming a color picture using the preservation stability or the obtained wet toner as a wet toner and color mixture nature are taken into consideration, as carbon black and a yellow system, it is desirable by the black system also in the above-mentioned coloring agent to use a copper phthalocyanine blue in brilliant carmine 6B and a cyanogen system by the mixture of benzidine yellow and Hansa yellow and the Magenta system.
[0023] Although the amount used can be chosen as arbitration in 0.0001 - 2000% of the weight of the range to resin weight, in order to reproduce a multicolor continuation gradient equivalent to offset printing, the optical reflection density after the imprint to the transferred object of each color toner is [0.7 or more] required for a certain thing, and a certain thing is [1.0 or more] desirable [a coloring agent is good to use the thing of the shape of powder with a particle size of 30-150 micrometers by the secondary state of aggregation, and / the amount used] about especially cyanogen and black. In order to make optical reflection density or more into 0.7 about each color, in the case of black and cyanogen, it is 10 - 150 % of the weight on the same weight criteria as the above, and it is good to consider [in the case of a Magenta] as 10 - 100 % of the weight 40 to 150% of the weight in the case of yellow. If the above-mentioned range is crossed also about which color, it will become easy to produce a greasing after development.
[0024] In non-subtlety particle fine particles, a silicon dioxide, an aluminum oxide, titanium dioxides, and those gelation objects, Viscosity minerals which use an aluminum oxide and silicon oxide as a principal component, such as a kaolinite and a montmorillonite, are mentioned. For example, trade name AEROSIL 130 and AEROSIL 200 and AEROSIL200 -- CF, AEROSIL300, and AEROSIL 300CF and AEROSIL 380 and AEROSIL OX50 and AEROSIL TT600P and AEROSIL MOX80 -- AEROSIL MOX170, AEROSIL COK84, AEROSILR972, AEROSIL R974, AEROSIL R202, AEROSIL R805, AEROSIL R812 and Aluminium Oside C, Titanium Dioxide P25, (the product made from Japanese Aerosil), Idemitsu titania IT-S, Idemitsu titania IT-OA, Idemitsu titania IT-alumnus, Idemitsu titania IT-OC, Idemitsu titania IT-OD5, Idemitsu titania IT-OD10, Idemitsu titania IT-OD15, Idemitsu titania IT-OD20, Idemitsu titania IT-WA, Idemitsu titania IT-WB, Idemitsu titania IT-PA, Idemitsu titania IT-PB, Idemitsu titania IT-PC, Idemitsu titania IT-DA, Idemitsu titania IT-DB, Idemitsu titania IT-DC, Idemitsu titania IT-DD, Idemitsu titania IT-DE, Idemitsu titania IT-UDA, Idemitsu titania IT-UDB (Idemitsu Kosan make), a bentonite (product made from Pure Chemistry), a kaolin (product made from Pure Chemistry), etc. are mentioned.
[0025] The big specific surface area worked effectively, showed high adsorption capacity, and heightened the cohesive force of a toner particle greatly, and these non-subtlety particle fine particles have prevented image generating flow. Although the addition of non-subtlety particle fine particles changes with the diameter of a primary particle, and specific surface area, it usually adds 0.01 % of the weight - 100 % of the weight to toner solid content. Moreover, non-subtlety particle fine particles may be two or more kinds of mixture. Moreover, if a thing 10 micrometers or less has a desirable particle size and non-subtlety particle fine particles become larger than 10 micrometers, they will have a bad influence on image quality in respect of definition, a rough deposit, etc. Moreover, during an olefin system resin solution, coloring agent dispersion liquid, those mixed liquor, and cooling granulation, although non-subtlety particle fine particles may add the solvent of olefin system resin in process [any after the permutation by liquefied aliphatic hydrocarbon], adding after a solvent permutation is desirable.
[0026] Furthermore, to the wet toner of this invention, the polar liquid of amino alcohol, such as alcohols, such as toluene, a tetrahydrofuran and a methanol, ethanol, and propanol, and 3-pyridyl propanol, may be added 0.05 to 5% of the weight to a part for solid [of a wet toner]. Since dispersibility can be raised and a resin particle can be made to adsorb easily while a resin particle gets wet by this addition and raising a property, image quality and an electrification property are improvable. In addition, the alkyd resin which is not denaturalized [various meltable resin, for example, denaturation or], usual acrylic resin, synthetic rubber, polyalkylene oxide, a polyvinyl acetal, a polyvinyl butyral, vinyl acetate resin, etc. can be added for example, into an electric insulation liquid as a fixing agent.
[0027] Moreover, the synthetic resin which can add the surfactant of many anion systems, a cation

system, both sexes, or the Nonion system as a dispersant, and is used as the above-mentioned fixing agent can also be used as a dispersant.
[0028] Next, the manufacture approach of the wet toner of this invention is explained. In giving a mixed distribution process and manufacturing a wet toner, after mixing with liquefied aliphatic hydrocarbon and cooling subsequently under warming of the olefin system resin which have a carboxyl group or an ester group, and a coloring agent, the 1st manufacture approach of the wet toner of this invention be characterize by making the polyhydroxy carboxylate and lecithin of 3 - 10 **** which make hydroxycarboxylic acid ester a monomer exist in the production process.
[0029] Mixed distribution of the olefin system resin is thrown in and carried out under warming into liquefied aliphatic hydrocarbon. warming -- a general temperature requirement is 40 degrees C - 120 degrees C that for resin to plasticize, to be sufficient temperature to liquefy as conditions, and what is necessary is just the temperature requirement which each component does not decompose. The rate of olefin system resin to liquefied aliphatic hydrocarbon should just be the range used as the letter of a flow, in case mixed distribution is carried out under warming of olefin system resin in liquefied aliphatic hydrocarbon.
[0030] Moreover, apart from this resin dispersant, a coloring agent is thrown in in liquefied aliphatic hydrocarbon, it mixes, and coloring agent dispersion liquid are prepared. A coloring agent is good to make it distribute in liquefied aliphatic hydrocarbon so that it may become a predetermined mixing ratio to resin.
[0031] The prepared coloring agent dispersion liquid are cooled, after supplying at a time in the above-mentioned resin dispersant and carrying out churning mixing in a 40 degrees C - 120 degrees C temperature requirement. Even if it quenches cooling using dry ice, liquid nitrogen, etc., you may supply in the cooled liquefied aliphatic hydrocarbon, natural radiationnal cooling may be carried out, for example, it is cooled by 5-15 degrees C, and a resin particle precipitates from dispersion liquid during cooling.
[0032] Ultrasonic irradiation, a high speed DISU parser, a jet mill, NIBURA, on-GUMIRU, a ball mill, an atomizer, etc. can perform the mixed distributed-processing process performed after cooling, and, thereby, it can atomize a resin particle further. That is, what shows the single peak whose mean particle diameter the range of the particle size of the particle obtained is 0.1-10 micrometers, and is 0.6-0.8 micrometers is obtained. Moreover, if mealing only of the coloring agent is first carried out at another process, it can consider as a wet toner with a still more sharp particle size.
[0033] The resin particle obtained by this invention has the narrow distribution width of face of the particle size, and what shows a single peak is obtained. Although this detailed reason is unknown, it is thought that it is based on the granulation adjustment function and distributed function of polyhydroxy carboxylate in the granulation process of a resin particle.
[0034] Although polyhydroxy carboxylate is good to add in pigment dispersion liquid, it may be added at the phase which could add in the resin dispersant and mixed pigment dispersion liquid and a resin dispersant, and the distributed process after cooling.
[0035] Next, the 2nd manufacture approach of the wet toner of this invention is explained. It is characterized by to permute a solvent with this aliphatic hydrocarbon while supplying , cooling this resin solution the bottom of the polyhydroxy carboxylate existence of 3 which makes hydroxycarboxylic acid ester a monomer - 10 **** , and in liquefied aliphatic hydrocarbon and depositing a resin particle , after using the 2nd manufacture approach of this invention as the resin solution which carried out the heating dissolution of olefin system resin and the coloring agent in the solubility over this resin at the high solvent of temperature dependence , and the coloring agent distributed .
[0036] the solvent to olefin system resin -- resin -- warming -- each solubility difference (25 degrees C and 65 degrees C) should just be a thing 0.05g / more than Solvent ml preferably more than 0.01g / solvent ml that that you make it insolubilize by dissolving and cooling in the thing which sometimes dissolves and is not dissolved in ordinary temperature, or ordinary temperature etc. should just be the solvent which gives temperature dependence to solubility. As such a solvent, a tetrahydrofuran, benzene, toluene, a xylene, dimethylformamide (DMF), dimethyl sulfo oxide (DMSO), an acetone, a methyl ethyl

ketone (MEK), etc. are mentioned, for example. The amount of dissolutions of the resin occupied to total of a solvent and resin is arbitrary, and is not cared about. However, when a resin ratio is too high, since there is a possibility that a resin particle may contact mutually and may serve as a gel lump in the deposit process of a resin particle, it is good to consider as 1 - 80% of the weight of the range. warming for dissolving resin -- as for conditions, warming beyond the need is not [that what is necessary is just the minimum temperature required for the dissolution of resin] desirable. warming -- conditions are the same as that of the manufacture approach of the above 1st. Agitating according to the usual approach is desirable during the dissolution.

[0037] So that the thing of the shape of powder with a particle size of 30-150 micrometers may be used for a coloring agent by the secondary state of aggregation and it may become a predetermined mixing ratio about resin and a coloring agent After carrying out heating melting of the resin, mixing a coloring agent, making it distribute and dissolve into a solvent and making a solvent distribute resin and a coloring agent by the dissolution or ultrasonic distribution separately, you may mix and a powder coloring agent may be distributed in a resin solution. Moreover, if mealing only of the coloring agent is first carried out at another process, it can consider as a wet toner with a still more sharp particle size. You may add in a resin solution and polyhydroxy carboxylate may be added in pigment dispersion liquid, although you may add at a granulation process.

[0038] The granulation process of the resin particle in this invention is performed by supplying in the liquefied aliphatic hydrocarbon which cooled the resin solution. It is desirable to face to throw in a resin solution in liquefied aliphatic hydrocarbon, and to improve distribution of the resin particle which deposits desirable with distributed means, such as churning and/or ultrasonic irradiation. Even if it quenches using dry ice, liquid nitrogen, etc., cooling may be supplied to the cooled electric insulation liquid, and may carry out natural radiationnal cooling. Cooling conditions are the same as that of the manufacture approach of the above 1st.

[0039] If a resin solution is thrown in in liquefied aliphatic hydrocarbon, a deposit of a resin particle will produce it to resin according to a solubility difference with the liquefied aliphatic hydrocarbon which is a poor solvent in the deposit and coincidence of a resin particle by the temperature gradient of a resin solution. the thing of a submicron unit is obtained for the particle size, and it may be [the resin particle which deposits / particle size distribution] narrow. That is, although the particle size of the particle obtained does not especially need ball milling actuation of a resin particle in which were the range of 0.1-10 micrometers, and mean particle diameter showed the single peak which is 0.6-0.8 micrometers, and was needed by the conventional approach, a classification, etc., it can atomize a resin particle further by carrying out mixed distributed processing with ultrasonic irradiation, a high speed DISU parser, a jet mill, NIBURA, on-GUMIRU, a ball mill, an atomizer, etc.

[0040] If polyhydroxy carboxylate is made to exist in a granulation process, it will be thought that the distributed function to the inside of liquefied aliphatic hydrocarbon is achieved with the granulation adjustment function to the resin particle which deposits from dissolving into liquefied aliphatic hydrocarbon and having a resin particle and strong compatibility.

[0041] After depositing a resin particle, it is desirable to carry out the solvent permutation of the solvent with liquefied aliphatic hydrocarbon. It is good for means, such as standing or centrifugal separation, to separate and wash a deposit resin particle as the approach, to remove a solvent, and to distribute liquefied aliphatic hydrocarbon.

[0042] Although it is possible to perform the electric-field imprint of the usual imprint approach, i.e., a corona transfer etc., to insulating candidates for an imprint, such as paper, of course when using the wet toner by this invention as an object for electrophotography, it can imprint from the photo conductor front face for electrophotography efficiently with a pressure replica method to candidates for an imprint of electric conductivity, such as a metal.

[0043] Moreover, the streak section with high print durability can be formed in the monotonous printing version by imprinting the toner of this invention to the substrate for the monotonous printing versions. It thinks for it being thought that the adhesive property of this with paper, plastics, a metal, etc. improves by existence of the carbonyl group in the carboxyl group and ester group which are contained in the

resin particle in a toner, and the moderate flexibility brought about by existence of a resin particle to absorb the impact at the time of printing.
[0044]
[Function] The olefin system resin particle independence which has a synthetic-resin particle especially a carboxyl group, or an ester group, In the wet toner which made the polyhydroxy carboxylate of 3 which makes hydroxycarboxylic acid a monomer - 10 **** exist in the olefin system resin particle which has the carboxyl group or ester group which added the coloring agent, and liquefied aliphatic hydrocarbon Or silicon oxide, titanium oxide, Adsorption of the superfluous ion which is the wet toner which prevented image flow by having made non-subtlety particle fine particles, such as an aluminum oxide, exist, and exists in a wet toner Or it is concluded that image flow was prevented by the interaction with a pigment and a resin particle etc. when the particle cohesive force at the time of approach grouping in development increased.
[0045]
[Example]
Eve FREX 250 (ethylene-vinyl acetate copolymer, Made in [DEYUPON poly chemical] Mitsui) 2.5g, 1.0g [of tin octylate] (trade name: NIKKAOKU Chicks Sn, 28% [of metal content], Nihon Kagaku Sangyo Co., Ltd. make), and tetrahydrofuran 50ml was mixed in 1200ml eggplant mold flask of examples, it flowed back under the 75-degree C water bath for 1 hour, resin was dissolved, and the resin solution was prepared.
[0046] On the other hand, it is Seika Fast Yellow to another container. 2400B(Product made from formation of great Nissei) 2.5g, Polly 12-methyl-hydroxystearate ester (the product made from Ito Oil Mill --) Wax 90mg of a trimer, the acid numbers 40.8-42.8, saponification values 196.9-197.7, weight average molecular weight 1200, hues (Gardner HERIGE) 6-7, and light ashes brown, Mixed tetrahydrofuran 50ml, the ultrasonic homogenizer (made in NIPPON SEIKI Factory, US-300T) was made to distribute for 10 minutes, and pigment dispersion liquid were prepared.
[0047] After supplying in the resin solution which prepared these pigment dispersion liquid previously and flowing back under a further 70-80-degree C water bath for 1 hour, it supplied in Isopar G(product made from Exon Chemistry) 300ml ice-cooled beforehand, and the ultrasonic homogenizer distributed for 2 minutes. Furthermore, it evaporated and the evaporator separated the tetrahydrofuran from these dispersion liquid, and Isopar G was added so that it might become 1.5% of solid content concentration. When grading analysis was carried out for this wet toner with the micro truck IISRA mold (made in [slop company] Nikkiso Co., Ltd. and North), it had the distribution spectrum of a single peak by distribution width of face of 0.17-1.69 micrometers in Sharp whose mean particle diameter is 0.36 micrometers.
[0048] Furthermore, it is AEROSIL to wet toner 350g. 300 [20mg] (mean diameter: 7nm, specific surface area of 300m 2 / g, product made from Japanese Aerosil) was added, the ultrasonic homogenizer distributed for 1 minute, and the desired wet toner was obtained.
[0049] It is 237 in order to evaluate the wet toner prepared as mentioned above. High Voltage Source Measure Unit (product made from KEITHREY) was used and the initial current value and the current value of 60 seconds after were measured. Measurement filled the wet toner to the 5.0x4.5cm inter-electrode one made from brass placed in parallel with spacing of 1cm, and was performed by impressing the electrical potential difference of 1000V. Moreover, evaluation of image flow formed the various electrostatic patterns of surface charge 20-150V on electrostatic recording paper (for D-Scan electro static plotters made from SEIKO Electron), and evaluated by viewing the image which developed negatives with the roller developing machine and was obtained. An evaluation result is shown in Table 1.
[0050] AEROSIL in example 2 example 1 Except having set the addition of 300 to 40mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1.
[0051] Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face

of 0.17-1.69 micrometers and whose mean particle diameter are 0.48 micrometers. A logarithm expresses particle size by the axis of abscissa to drawing 1 , and the histogram and accumulation particle size distribution showing frequency are expressed with the polygonal line to an axis of ordinate.
[0052] AEROSIL in example 3 example 1 Except having set the addition of 300 to 240mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1.
[0053] Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-1.69 micrometers and whose mean particle diameter are 0.49 micrometers.
[0054] AEROSIL in example 4 example 1 It replaces with 300 and is AEROSIL. Except having used 200 (mean diameter: 12nm, specific-surface-area:200m2 / g, product made from Japanese Aerosil) 40mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1.
[0055] Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-2.63 micrometers and whose mean particle diameter are 0.60 micrometers.
[0056] Seika Fast Yellow FBR in example 5 example 1 2400B -- replacing with -- Monastral (the product made from ICI --) Blue Use a metal phthalocyanine pigment, and replace with tin octylate and JIISO sulfo succinic-acid cobalt 256mg is used. Moreover, AEROSIL It replaces with 300 and is AEROSIL. Except having used 40mg of 200, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1.
[0057] Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-3.73 micrometers and whose mean particle diameter are 0.54 micrometers. A logarithm expresses particle size by the axis of abscissa to drawing 2 , and the histogram and accumulation particle size distribution showing frequency are expressed with the polygonal line to an axis of ordinate.
[0058] Seika Fast Yellow in example 6 example 1 It replaces with 2400B and is MITSUBISHI. Carbon black MA-100 (Mitsubishi Kasei Corp. make) is used, and it replaces with tin octylate, and JIISO sulfo succinic-acid cobalt 256mg is used, and it is AEROSIL. Except having set the addition of 300 to 40mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1.
[0059] Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-1.69 micrometers and whose mean particle diameter are 0.53 micrometers.
[0060] It replaces with the tin octylate in example 7 example 1, and octylic acid zirconium (trade name: NIKKAOKU Chicks Zr, 4% [of metal content], Nihon Kagaku Sangyo Co., Ltd. make) 1.0g is used, and it is AEROSIL. It replaces with 300 and is AEROSIL. R-805 Except having been referred to as 160mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1. Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-5.27 micrometers and whose mean particle diameter are 1.12 micrometers.
[0061] AEROSIL in example 8 example 1 It replaces with 300 and is Aluminium. Oxide Except having used C(mean diameter: 20nm, specific surface area of 100m 2 / g, product made from Japanese Aerosil) 40mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1. Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-3.73 micrometers and whose mean particle diameter are 0.59 micrometers.
[0062] the tin octylate in example 9 example 1 -- replacing with -- an octylic acid zirconium (trade name: -- NIKKAOKU Chicks Zr --) 28% of metal content and 1.0by Nihon Kagaku Sangyo Co., Ltd. g

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are used, and it is AEROSIL IT-S (mean particle diameter: 17nm). It replaces with 300 and is the Idemitsu titania. specific surface area of 130m 2 / g, and the Idemitsu Kosan make -- except having used 40mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1. Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-1.69 micrometers and whose mean particle diameter are 0.62 micrometers.

[0063] the tin octylate in example 10 example 1 -- replacing with -- an octylic acid zirconium (trade name: -- NIKKAOKU Chicks Zr --) 28% of metal content and 1.0by Nihon Kagaku Sangyo Co., Ltd. g are used, and it is AEROSIL IT-PB (mean particle diameter: 40nm). It replaces with 300 and is the Idemitsu titania. specific surface area of 220m 2 / g, and the Idemitsu Kosan make -- except having used 40mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1. Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the distribution spectrum of a single peak in Sharp distribution width of face of 0.17-3.73 micrometers and whose mean particle diameter are 0.62 micrometers.

[0064] It replaces with the tin octylate in example 11 example 1, and octylic acid zirconium (trade name: NIKKAOKU Chicks Zr, 28% [of metal content], Nihon Kagaku Sangyo Co., Ltd. make) 1.0g is used, and it is AEROSIL. Except having replaced with 300 and having used bentonite (product made from Pure Chemistry) 40mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1.

[0065] Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-2.63 micrometers and whose mean particle diameter are 0.70 micrometers. A logarithm expresses particle size by the axis of abscissa to drawing 3 , and the histogram and accumulation particle size distribution showing frequency are expressed with the polygonal line to an axis of ordinate.

[0066] It replaces with the tin octylate in example 12 example 1, and octylic acid zirconium (trade name: NIKKAOKU Chicks Zr, 28% [of metal content], Nihon Kagaku Sangyo Co., Ltd. make) 1.0g is used, and it is AEROSIL. Except having replaced with 300 and having used kaolin (product made from Pure Chemistry) 40mg, a wet toner is prepared like an example 1 and the result of having evaluated the toner property like the example 1 is shown in Table 1.

[0067] Moreover, when grading analysis was carried out like the example 1 about the wet toner after a solvent permutation, it had the spectrum distribution of a single peak in Sharp distribution width of face of 0.17-1.69 micrometers and whose mean particle diameter are 0.61 micrometers.

[0068] Eve FREX 250 (ethylene-vinyl acetate copolymer, Made in [DEYUPON poly chemical] Mitsui) 2.5g, 1.0g [of tin octylate], and tetrahydrofuran 50ml was mixed in 13200ml eggplant mold flask of examples, it flowed back under the 70-80-degree C water bath for 1 hour, resin was dissolved, and the resin solution was prepared.

[0069] On the other hand, it is Seika Fast Yellow to another container. 2400B(Product made from formation of great Nissei) 2.5g, Polly 12-methyl-hydroxystearate ester (the product made from Ito Oil Mill --) Wax 90mg of a trimer, the acid numbers 40.8-42.8, saponification values 196.9-197.7, weight average molecular weight 1200, hues (Gardner HERIGE) 6-7, and light ashes brown, AEROSIL Mixed 300 (mean diameter: 7nm, specific surface area of 300m 2 / g, product made from Japanese Aerosil), and tetrahydrofuran 50ml, the ultrasonic homogenizer (made in NIPPON SEIKI Factory, US-300T) was made to distribute for 10 minutes, and pigment dispersion liquid were prepared.

[0070] After supplying in the resin solution which prepared these pigment dispersion liquid previously and flowing back under a further 70-80-degree C water bath for 1 hour, it supplied in Isopar G(product made from Exon Chemistry) 300ml ice-cooled beforehand, and the ultrasonic homogenizer distributed for 2 minutes. Furthermore, it evaporated and the evaporator separated the tetrahydrofuran from these dispersion liquid, Isopar G was added and the desired wet toner was obtained so that it might become 1.5% of solid content concentration.

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[0071] When grading analysis of this wet toner was carried out, it had the spectrum distribution of a single peak by distribution width of face of 0.17-1.69 micrometers in Sharp whose mean particle diameter is 0.50 micrometers. Moreover, the result of having evaluated like [toner / this / wet] the example 1 is shown in Table 1.

[0072] They are Eve FREX 250 (ethylene-vinyl acetate copolymer, Made in [DEYUPON poly chemical] Mitsui) 2.5g, 1.0g of tin octylate, and AEROSIL in 14200ml eggplant mold flask of examples. 300 (mean diameter: 7nm, specific-surface-area 300m2/g, product made from Japanese Aerosil) tetrahydrofuran 50ml was mixed, it flowed back under the 70-80-degree C water bath for 1 hour, resin was dissolved, and the resin solution was prepared.

[0073] On the other hand, it is Seika Fast Yellow to another container. 2400B(Product made from formation of great Nissei) 2.5g, Polly 12-methyl-hydroxystearate ester (the product made from Ito Oil Mill --) Wax 90mg of a trimer, the acid numbers 40.8-42.8, saponification values 196.9-197.7, weight average molecular weight 1200, hues (Gardner HERIGE) 6-7, and light ashes brown, Mixed tetrahydrofuran 50ml, the ultrasonic homogenizer (made in NIPPON SEIKI Factory, US-300T) was made to distribute for 10 minutes, and pigment dispersion liquid were prepared.

[0074] After supplying in the resin solution which prepared these pigment dispersion liquid previously and flowing back under a further 70-80-degree C water bath for 1 hour, it supplied in Isopar G(product made from Exon Chemistry) 300ml ice-cooled beforehand, and the ultrasonic homogenizer distributed for 2 minutes. Furthermore, it evaporated and the evaporator separated the tetrahydrofuran from these dispersion liquid, Isopar G was added so that it might become 1.5% of solid content concentration, and the desired wet toner was obtained.

[0075] When grading analysis of this wet toner was carried out, it had the spectrum distribution of a single peak by distribution width of face of 0.17-1.69 micrometers in Sharp whose mean particle diameter is 0.65 micrometers. A logarithm expresses particle size by the axis of abscissa to drawing 4 , and the histogram and accumulation particle size distribution showing frequency are expressed with the polygonal line to an axis of ordinate. Moreover, the result of having evaluated like [toner / this / wet] the example 1 is shown in Table 1.

[0076] Eve FREX 250 (ethylene-vinyl acetate copolymer, Made in [DEYUPON poly chemical] Mitsui) 2.5g and lecithin (product made from From Soy Beans Pure Chemistry) 500ml were mixed in 15200ml eggplant mold flask of examples, heating stirring was carried out under the 120-degree C oil bath for 1 hour, resin was dissolved, and the resin solution was prepared.

[0077] container another on the other hand -- Monastral FBR Blue (the product made from ICI --) 2.5g of metal phthalocyanine pigments, and Polly 12-methyl-hydroxystearate ester (the product made from Ito Oil Mill --) Wax 90mg of a trimer, the acid numbers 40.8-42.8, saponification values 196.9-197.7, weight average molecular weight 1200, hues (Gardner HERIGE) 6-7, and light ashes brown, Mixed Isopar G100ml, the ultrasonic homogenizer (made in NIPPON SEIKI Factory, US-300T) was made to distribute for 10 minutes, and pigment dispersion liquid were prepared.

[0078] Isopar G beforehand ice-cooled after supplying in the resin solution which prepared these pigment dispersion liquid previously and carrying out heating stirring under a 120 more-degree C oil bath for 1 hour It supplied in 200ml and the ultrasonic homogenizer distributed for 2 minutes, and it diluted with Isopar G so that it might become 1.5% of solid content concentration further.

[0079] When grading analysis of this wet toner was carried out, it had the spectrum distribution of a single peak by distribution width of face of 0.17-5.27 micrometers in Sharp whose mean particle diameter is 1.35 micrometers. A logarithm expresses particle size by the axis of abscissa to drawing 5 , and the histogram and accumulation particle size distribution showing frequency are expressed with the polygonal line to an axis of ordinate. Furthermore, it is AEROSIL to wet toner 350g. 300 [40mg] (mean diameter: 7nm, specific surface area of 300m 2 / g, product made from Japanese Aerosil) was added, the ultrasonic homogenizer distributed for 1 minute, and the desired wet toner was obtained. Moreover, the result of having evaluated like [toner / this / wet] the example 1 is shown in Table 1.

[0080] It replaces with the Polly 12-methyl-hydroxystearate ester (trimer) in example 16 example 15. Polly 12-methyl-hydroxystearate ester (the product made from Ito Oil Mill --) The wax of a tetramer, the

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acid numbers 40.8-42.8, saponification values 196.9-197.7, weight average molecular weight 1200, hues (Gardner HERIGE) 6-7, and light ashes brown is used. Moreover, AEROSIL It replaces with 300, 40mg of Idemitsu titania ID-OA is used, a wet toner is prepared similarly, and the result of having evaluated like the example 1 is shown in Table 1. Moreover, when grading analysis of a wet toner was carried out like the example 1, it had the spectrum distribution of a single peak by distribution width of face of 0.17-7.46 micrometers in Sharp whose mean particle diameter is 1.17 micrometers.

[0081] It sets in the example of comparison 1 example 1, and is AEROSIL. Except not adding 300, a wet toner is prepared like an example 1 and the result of having evaluated like the example 1 is shown in Table 1.

[0082] It replaces with the tin octylate in example of comparison 2 example 1, and octylic acid zirconium 1.0g is used, and it is AEROSIL. Except not adding 300, a wet toner is prepared like an example 1 and the result of having evaluated like the example 1 is shown in Table 1.

[0083] It sets in the example of comparison 3 example 15, and is AEROSIL. Except not adding 300, a wet toner is prepared like an example 15 and the result of having evaluated like the example 1 is shown in Table 1.

[0084]

[Table 1]

表1

	初期電流値	６０秒後の電流値	画像流れ
実施例１	１６６８	１２６２	○
実施例２	１４４０	９７６	◎
実施例３	１６１９	８１３	◎
実施例４	１４２０	１１９７	◎
実施例５	１３８９	５２３	◎
実施例６	２７２８	２１０３	○
実施例７	３５６	２２３	◎
実施例８	８３９	６９８	◎
実施例９	３３２	２４９	◎
実施例１０	３７１	３０３	◎
実施例１１	４０５	２９２	◎
実施例１２	４１５	２７８	◎
実施例１３	１３８０	５２３	◎
実施例１４	１４３５	５７６	◎
実施例１５	１１８０	５２６	◎
実施例１６	１０４５	４８２	◎
比較例１	４８５	３７０	×
比較例２	１６０２	１３７１	×
比較例３	１３４５	４１０	×

ただし、◎：優、○：良、×：不良を示す。

[0085]

[Effect of the Invention] This invention by adding non-subtlety particle fine particles, such as silicon oxide, titanium oxide, and an aluminum oxide, in the wet toner which made the resin particle which added independence or a coloring agent for the synthetic-resin particle exist in liquefied aliphatic hydrocarbon The olefin system resin particle independence which prevents image flow and has especially a carboxyl group or an ester group, Or in the wet toner which made the polyhydroxy carboxylate of 3 which makes hydroxycarboxylic acid a monomer - 10 **** exist in the olefin system resin particle which has the carboxyl group or ester group which added the coloring agent, and a great portion of liquefied aliphatic hydrocarbon, it is effective.

[Translation done.]

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